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PROTECTIVE COATINGS
FOR REFRACTORY METALS
IN ROCKET ENGINES

Submitted to:

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Washington 25, D. C.
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Contract No. NAS7-113

PROTECTIVE COATINGS
FOR REFRACTORY METALS
IN ROCKET ENGINES

IITRI-B237-25
(Supplemental Report)
March 8, 1963 to March 15, 1964

Submitted to:

Chief, Liquid Propulsion Systems, Code RPL
National Aeronautics and Space Administration
400 Maryland Avenue, S. W.
Washington 25, D. C.

Attention: Mr. Henry Burlage, Jr.

March 18, 1965

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PROTECTIVE COATINGS
FOR REFRACTORY METALS
IN ROCKET ENGINES

I. INTRODUCTION

This report describes the results of a rocket engine test firing program designed to evaluate the performance of protective coating systems which were developed under Contract NAS7-113 during the period March 8, 1963, to March 15, 1964. The coating development effort that led to the selection of materials for evaluation has been previously reported.⁽¹⁾ A total of 13 nozzles, all based on tungsten, were evaluated. The coating systems consisted of silicides, silver-silicide mixtures, hafnium nitride, W-Hf alloy, and graded HfO_2 - SrZrO_3 -W composite coatings.

The firing tests were performed by Thiokol Chemical Corporation, Reaction Motors Division,⁽²⁾ using a thrust chamber specifically adapted to accommodate the nozzles. A mixture of 50 w/o N_2H_4 and UDMH fuel was used with N_2O_4 oxidizer operating at an O/F (oxidizer/fuel) ratio of 1.6. The nozzles were fired at sea level at a chamber pressure of 150 psia. The firing conditions were established to produce a wall temperature of approximately 4200° F at the surface of the coatings.

II. TEST PROGRAM

A. Facility

Figure 1 is a plot of c^* and flame temperature as a function of O/F for the engine employed. Table I provides data on combustion products at O/F = 1.5 and 1.7. Figure 2 is a schematic representation of the fluid supply system. Figure 3 is a photograph of the test installation with the thrust chamber installed. Supplementing the normal fluid system, a nitrogen constant bleed was provided between the nozzle and nozzle shroud to prevent oxidation. This system was originally sized to provide 500 cc/min STP

(1) "Protective Coatings for Refractory Metals in Rocket Engines," Contract NAS7-113, IITRI Report B237-25, August 31, 1964.

(2) "Test Evaluation of Coated Refractory Metal Nozzles in Rocket Engines," Contract NAS7-113, Report RMD 8997-F, Report period July 16, 1964 to September 21, 1964.

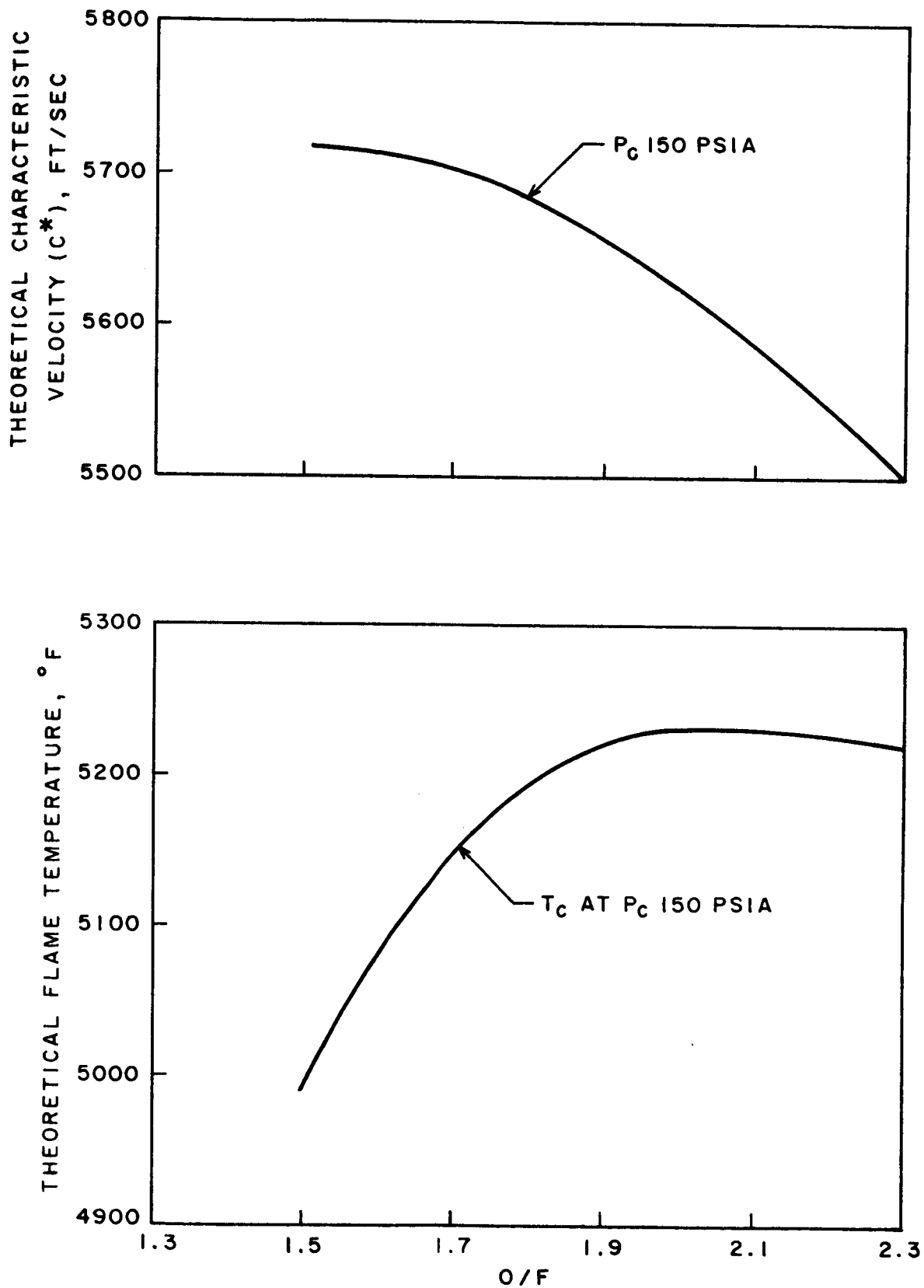


FIG. 1 - c^* AND FLAME TEMPERATURE VS. O/F FOR TEST ENGINE.

TABLE I

MOLE FRACTION OF CONVERGENT NOZZLE PRODUCTS

AT $P_c = 105$ PSIA

Combustion Products	Oxidizer/Fuel Ratio	
	1.5	1.7
CO	0.10409	0.09119
CO ₂	0.02995	0.03865
H	0.02166	0.02364
OH	0.01573	0.02751
H ₂	0.16112	0.11445
NO	0.00238	0.00528
O ₂	0.00112	0.00440
N ₂	0.32289	0.32821
O	0.00132	0.00347
H ₂ O	0.33970	0.36317

Propellants: Fuel - 50 w/o Hydrazine-UDMH
Oxidizer - N₂O₄

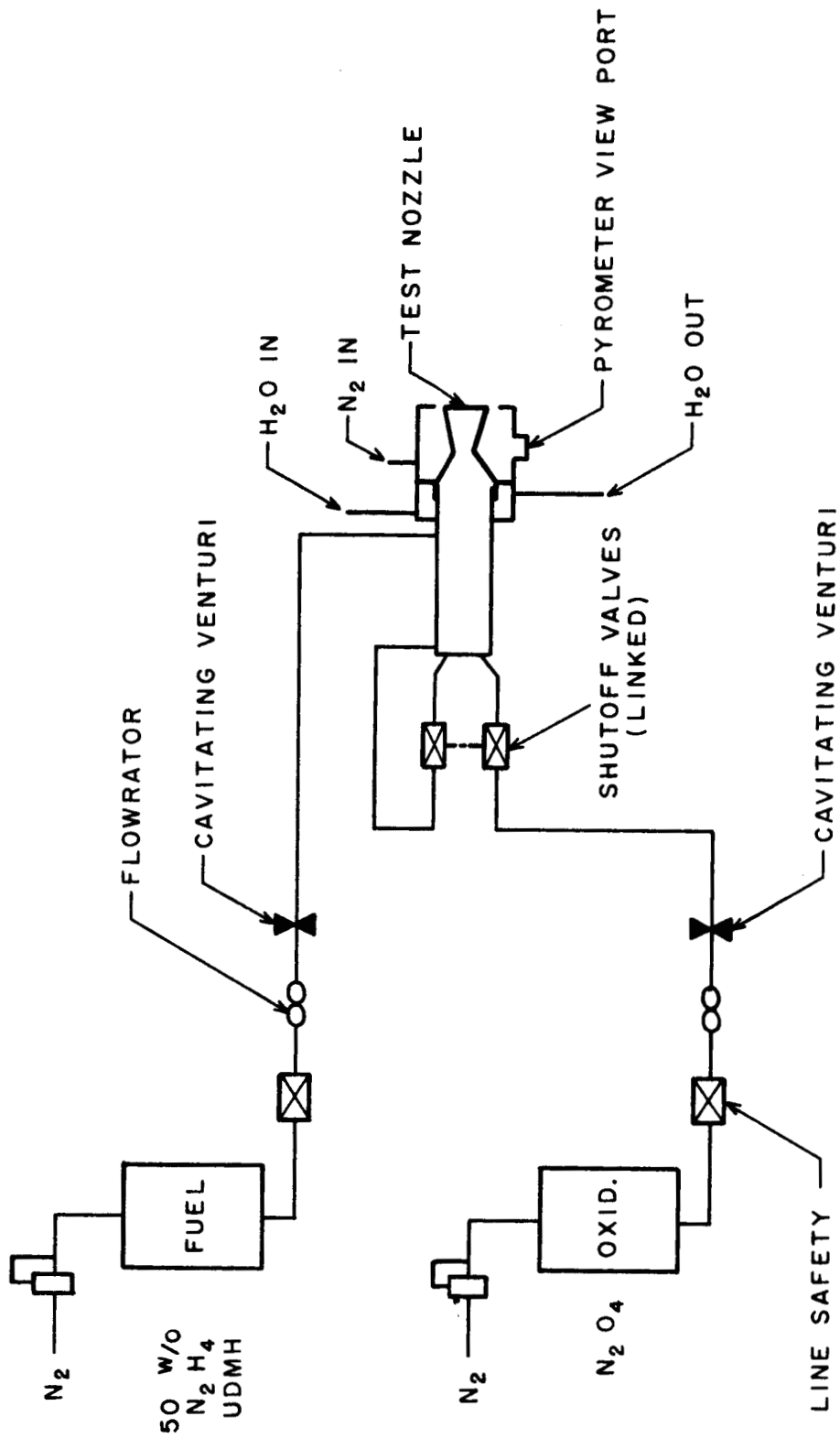


FIG. 2 - SCHEMATIC OF FLUID SUPPLY SYSTEM.

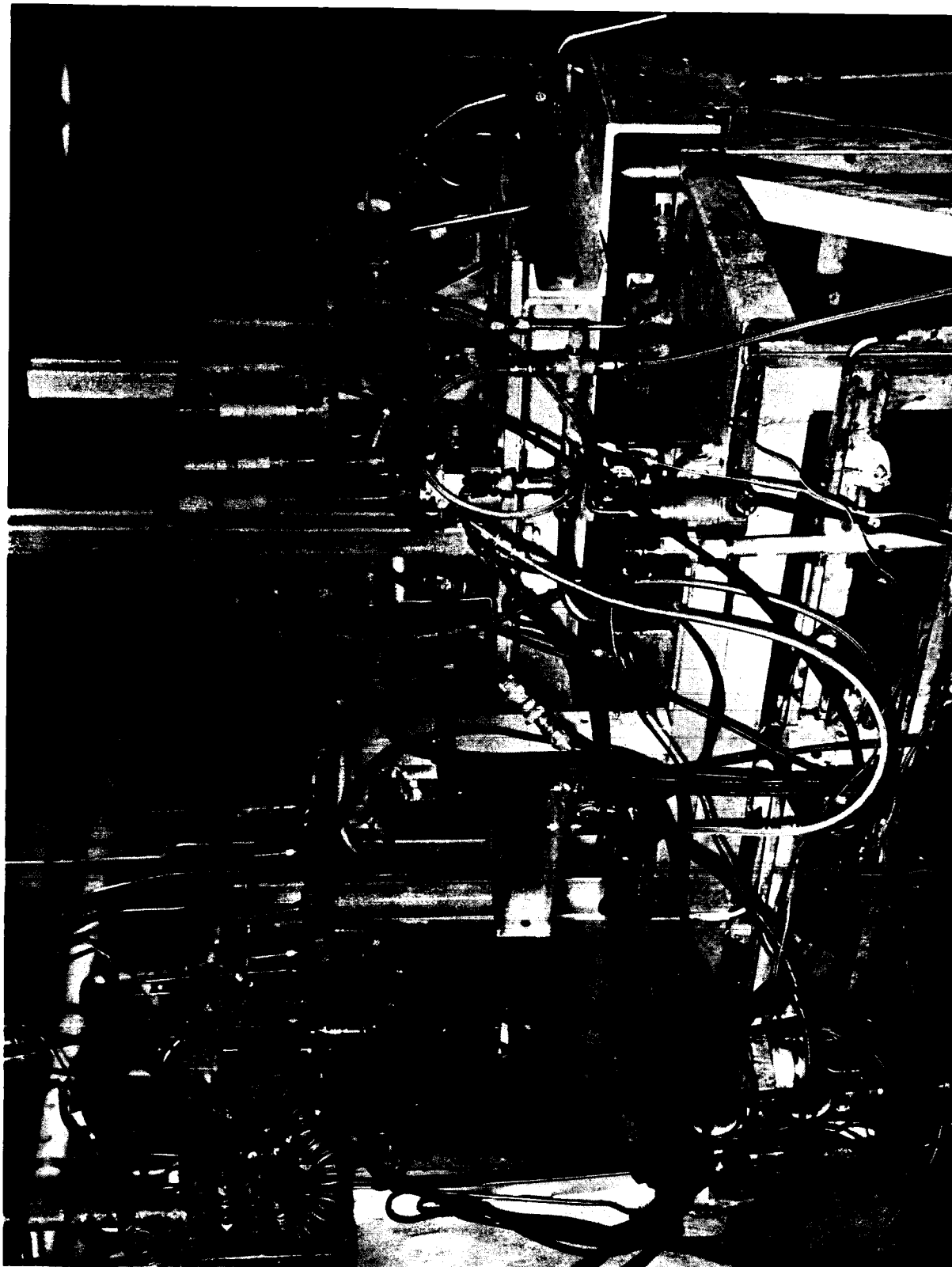


Fig. 3 - Photograph of Test Installation Showing Thrust Chamber and Test Nozzle with Gas Shroud Removed.

at 140 psia. When the quartz viewing port was removed, after run 5AX5493, the gas flow rate was increased to 3000 cc/min STP at 140 psia. Figure 4 is a closeup photograph showing the gas shroud installed. All of the nozzles that were tested were similar in configuration. The units were essentially sea level configurations with the following nominal dimensions:

$$D_t = 0.843 \text{ in.}$$

$$D_e = 1.50 \text{ in.}$$

$$1/2 \text{ Convergence angle} = 30^\circ$$

$$1/2 \text{ Divergence angle} = 15^\circ$$

The fabrication history of the nozzles was previously described.⁽¹⁾

Table II provides weight and actual dimensional data for each nozzle.

B. Discussion of Tests

Preliminary test firings were run to establish the adequacy of the nozzle adapter design and the general performance level for the modified thrust chamber. For these tests on Inconel X nozzle, coated with 0.018 in. Rokide Z on the ID surface, was used and operations were limited to 3 sec durations. The Inconel X nozzle test firings showed that a high c^* performance (96% of theoretical) could be anticipated during evaluation of the nozzles.

Table III presents a summary of all of the test results obtained on the nozzles. The first test nozzle (designation A-8, pack-siliconized tungsten) was evaluated during run 5AX5491. The purpose of this test was to determine the life of the best prior state-of-the-art coating system when tested under these conditions, and to further define the engine operating conditions. Test operations were normal in all respects. Progressive erosion of the throat was observed 3 sec after the operation began and convergent nozzle failure was incurred 11.8 sec after the start. Shutdown was signaled at this time; however, the water-cooled gas shroud shown in Figure 5 was damaged by the locally concentrated, high-velocity, hot gas stream. After reviewing the failure of the water-cooled shroud it was decided to conduct future tests with an uncooled graphite liner, 0.06 in. in thickness. Figure 6 shows the graphite liner installed in the jacket shroud which was run without coolant. Use of the graphite liner was successful, and failure of subsequent test nozzles did not damage the graphite. Also, a

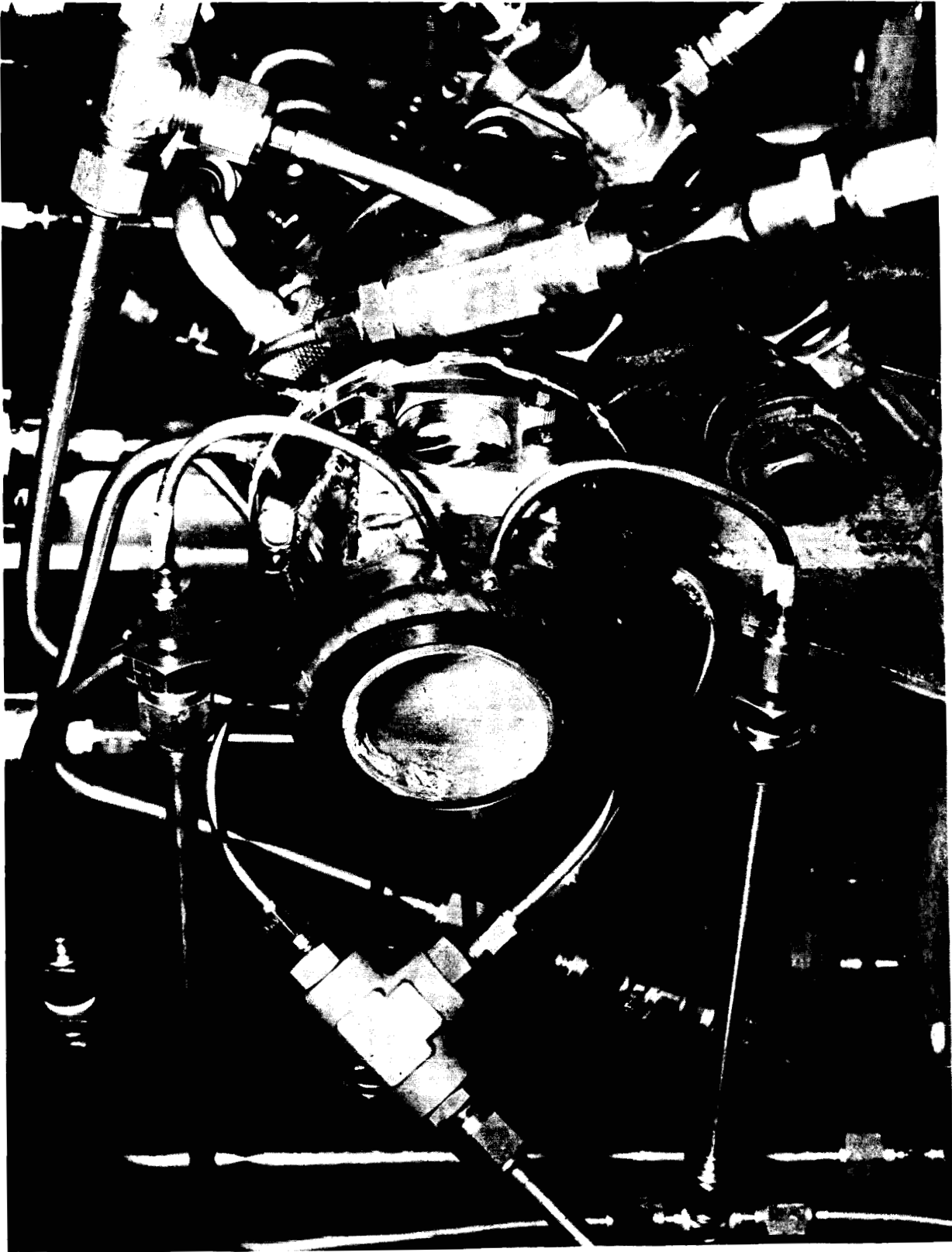


Fig. 4 - Close-up View of Showing Installed Gas Shroud.

TABLE II

NOZZLE WEIGHT AND DIMENSIONAL CHARACTERISTICS

Nozzle Designation	Weight, g	Dimensions (inches)					
		Convergent Cone Entry Plane		Divergent Cone Exit Plane		Throat	
		OD	ID	OD	ID	OD	ID
A-8	268	2.138	1.991	1.653		--	0.826/0.825
A-7	264	2.139	2.000	1.637	1.520	--	0.837
A-5	181	2.139	2.020	1.622	1.505	--	0.820/0.811
			1.926	1.594	1.478		
A-6	199	2.139	1.975	1.614	1.503	--	0.818/0.814
			1.920	1.591	1.488		
A-9	296	2.139	--	--	--	--	0.829
A-3	282	2.185	2.006	1.648	--	1.030	0.837/0.835
			1.988				
A-1	391	2.189	2.022	1.691	1.506	1.148	0.837/0.835
			1.987				
C-9	143	2.185	2.010	1.603	1.518	0.960	0.844/0.843
A-4	327	2.185	2.032	1.602	1.478	1.070	0.842/0.837
			1.983	1.587			
C-7	277	2.267	2.016	1.621	1.506	1.040	0.826/0.825
				1.614			
C-12	314	2.267	2.000	1.652	--	1.020	0.828/0.826
A-2	434	2.267	2.010	1.665	1.502	1.135	0.832/0.831
			2.006	1.648			
C-11	272	2.267	2.016	1.662	1.508	1.010	0.828

TABLE III

SUMMARY OF REFRACTORY NOZZLE COATING TEST RESULTS

Nozzle Designation	Coating and Nozzle Material	Run No. 5AX	c*, ft/sec	O/F	P _c , psia	Flame Temp., °F	Average Erosion Rate, mils/sec	Failure Time, sec	Failure Mode
A-8	Pack-siliconized wrought W	5491	5490	1.56	148.6	4610	2.64	11.8	(a)
A-7	Ag-Si on wrought W	5492	5410	1.62	148.6	4570	2.64	10.0	(a)
A-5	Ag-Si on plasma sprayed W	Cracked prior to testing, approximately 1 hr after shrink assembly							Fracture
A-6	(W-Hf)Si on plasma sprayed W	5493	--	--	93	--	None	0.06	Fracture
A-9	W ₂ Hf on wrought W	5494	5510	1.51	143.1	4770	1.3	20.5	(a)
A-3	W ₂ Hf on plasma sprayed W	5496	5400	1.58	148.4	4380	None	4.0	Fracture
A-1	HfN, W ₂ Hf on plasma-sprayed W	5497	5395	1.64	148.6	4410	None	2.1	Fracture
C-9	HfO ₂ , 60/40 HfO ₂ -W on plasma sprayed W, Mo	5498	--	--	150	--	None	0.057	Fracture
A-4	W ₂ Hf on plasma sprayed W	5500 ^(b)	5500	1.625	151.3	4680	--	4	Fracture

TABLE III (cont.)

Nozzle Designation	Coating and Nozzle Material	Run No. 5AX	c*, (ft/sec)	O/F	P _c ' psia	Flame Temp., °F	Average Erosion Rate, mils/sec	Failure Time, sec	Failure Mode
C-7	HfO ₂ , 40/60 HfO ₂ -W, 60/40 HfO ₂ -W on plasma sprayed W	5501	--	--	150	--	None	0.150	Fracture
C-12	HfO ₂ , 40/60 HfO ₂ -W on plasma sprayed W	5502	5500	1.62	150	4690	None	0.135	Fracture
A-2	Pack siliconized plasma sprayed W	5503 ^(c)	5480	1.66	152.4	4645	None	NA	NA
C-11	50/50 SrZrO ₂ -HfO ₂ , 60/40 HfO ₂ -W on plasma sprayed W	5505	5510	1.66	152.4	4730	2	9.1	Erosion
		5504	5540	1.66	152.4	4770	Not Est.	4.5	Coating spalled followed by rapid base metal erosion.

- (a) Coating melted, followed by rapid erosion of base metal.
 (b) Dual start employed for this nozzle. First start run duration 0.5 sec.
 (c) Run prematurely terminated after 3 sec.

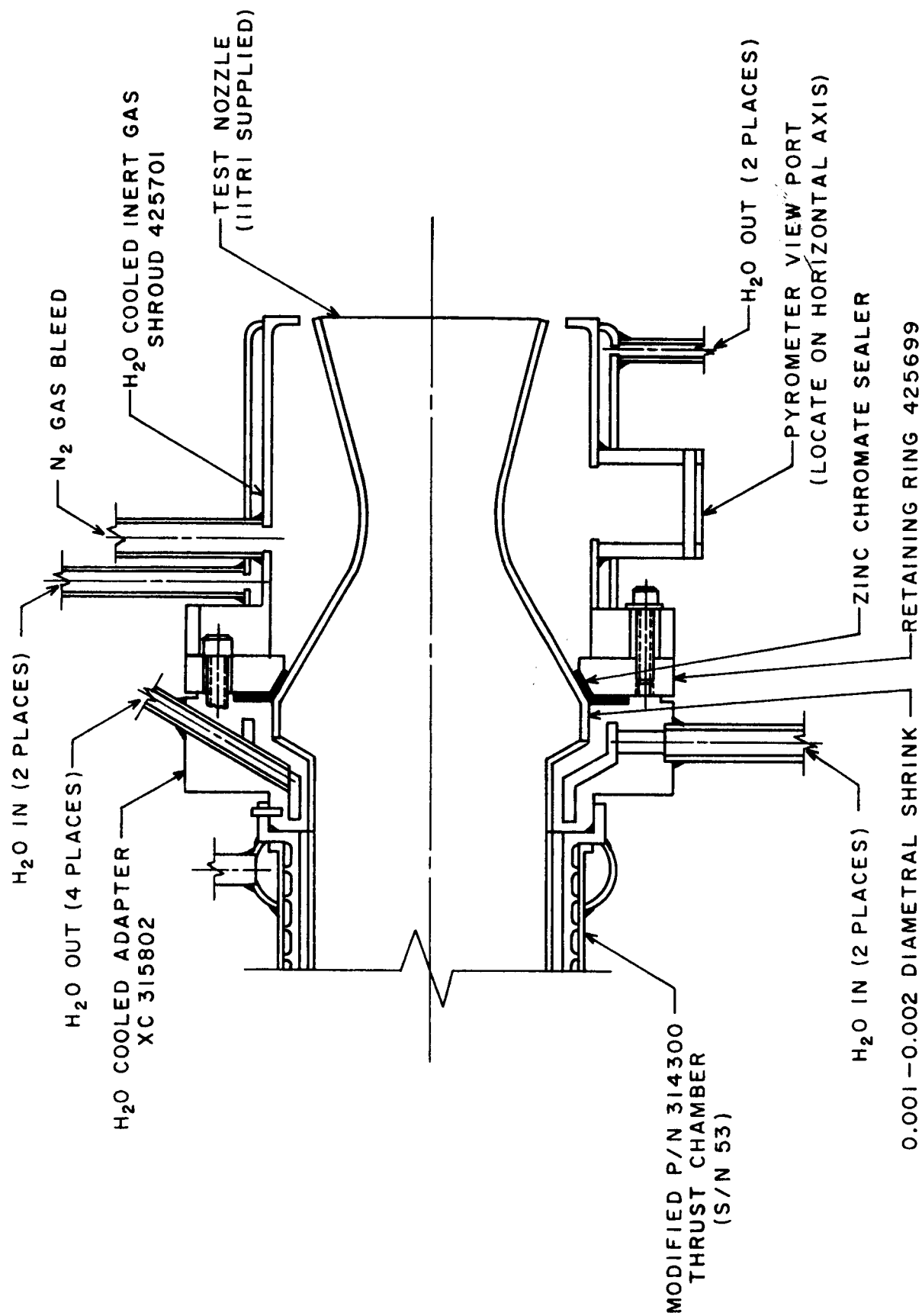


Fig. 5 - Test Installation for Refractory Metal Coated Nozzles (H₂O Cooled Inert Gas Shroud)

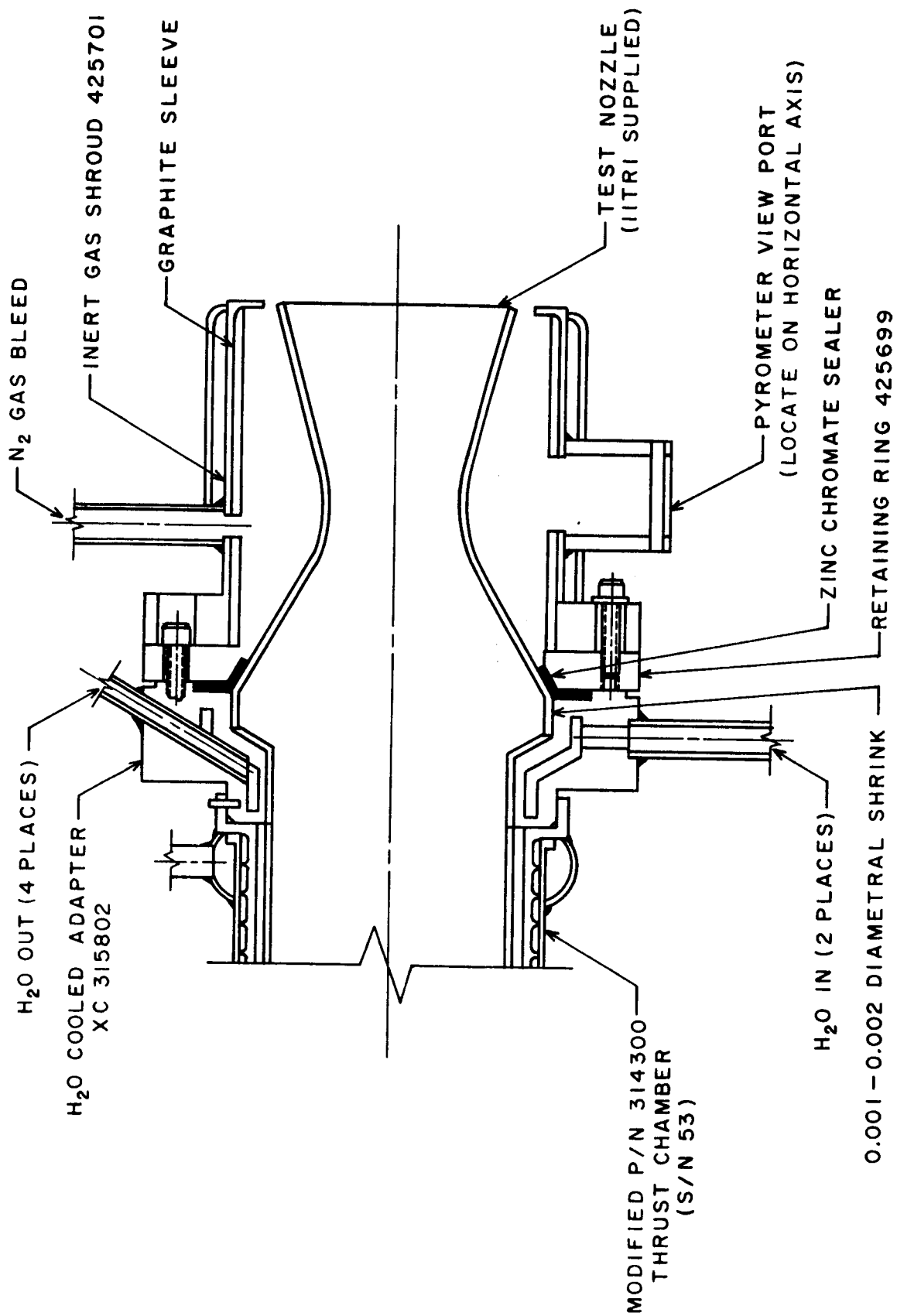


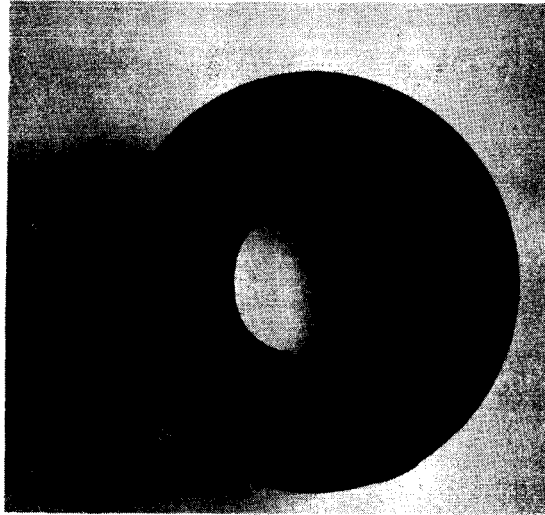
Fig. 6 - Test Installation for Refractory Metal Coated Nozzles (Uncooled Inert Gas Shroud)

heat transfer analysis showed that no significant reduction in radiant flux resulted from use of the graphite liner. Post-run examination of nozzle A-8 showed that melting had occurred almost throughout the entire convergent cone and throat areas of the nozzle. The erosion pattern may be seen in Figure 7. It may be noted that the most severe erosion and eventual burn-through occurred in the quadrant extending from 3 to 6 o'clock as viewed facing the injector. A similar failure was noted in the wrought tungsten nozzle A-7 (Figure 8), which had a Ag-Si slurry coating; however, the location of maximum erosion extended into the 6 to 9 o'clock quadrant as well.

One additional wrought tungsten nozzle, A-9, having a W_2Hf diffusion coating was evaluated. This nozzle showed a lower erosion rate and a longer time for the onset of erosion than the previous two nozzles. The throat erosion rate vs. time for these three nozzles may be seen in Figure 9. Evidence of localized throat erosion was observed in the W_2Hf coated nozzle extending for 270° , from the 12 to 9 o'clock positions. The remaining coating was oxidized and showed evidence of blistering beneath the oxide. This is the apparent result of volatile WO_3 formation.

The series of plasma-sprayed tungsten nozzles--A-1, A-3, A-4, A-5, A-6, C-7, C-9, and C-12--failed in thermal shock and could not be evaluated properly. One nozzle, A-5, failed after assembly into the adapter. A thermal stress analysis indicated that tensile stresses far in excess of the strength of tungsten were developed in a region of the convergent section slightly removed from the flange area. These stresses are apparently not relieved through plastic deformation and, hence, lead to the thermal shock failures observed.

To circumvent the thermal shock failure problems, several methods were investigated. Four nozzles (A-2, C-7, C-11, and C-12) were stress relief annealed in argon at $2050^\circ \pm 50^\circ F$ in an attempt to develop some degree of ductility in the tungsten. However, this was ineffective, as demonstrated by the subsequent thermal shock failure of nozzles C-7 and C-12.



Neg. No. 28128

**Fig. 7 - View of Convergent Section
of Nozzle A-8 After Test
Firing.**



Neg. No. 28129

**Fig. 8 - View of Convergent Section
of Nozzle A-7 After Test
Firing.**

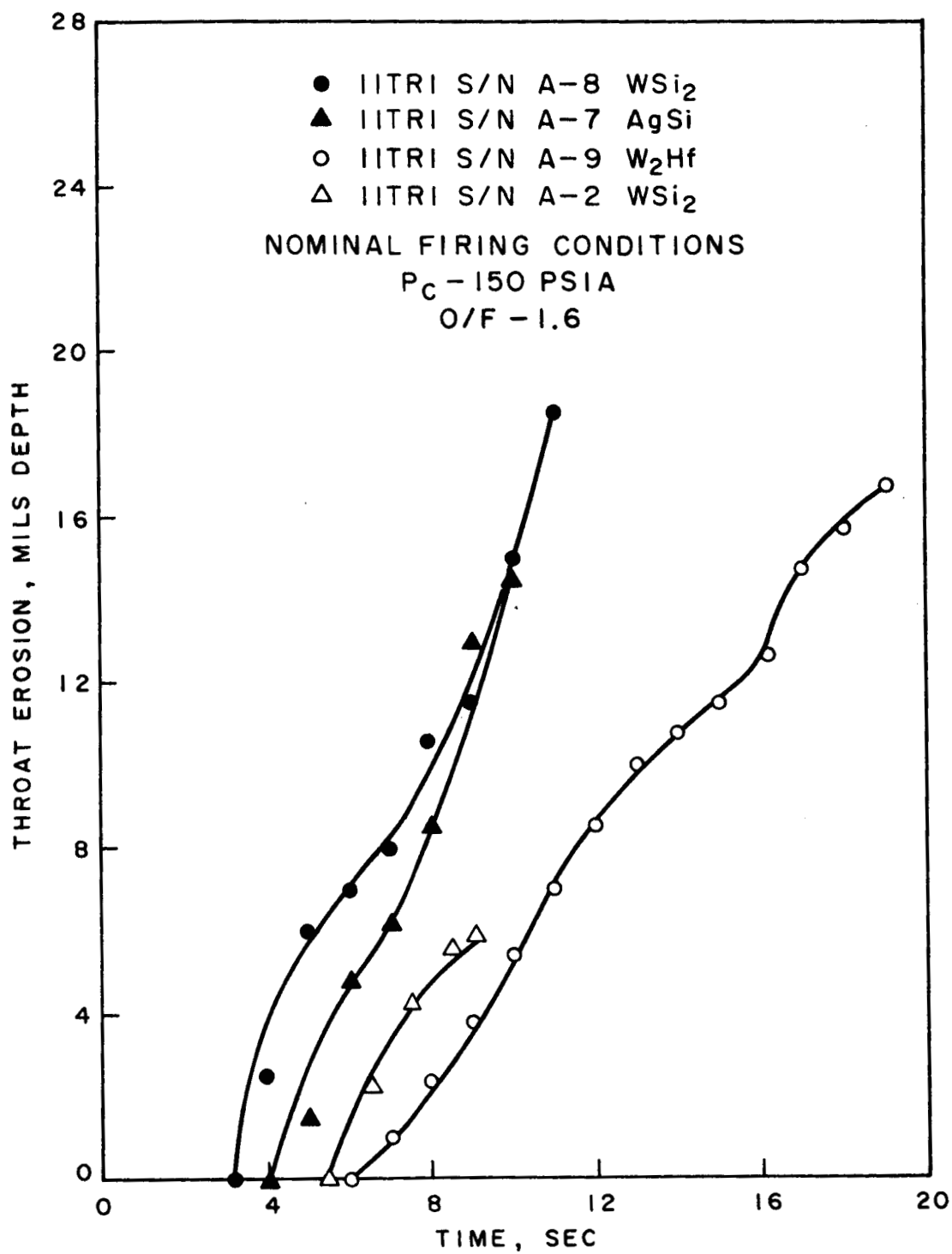


Fig. 9 - Throat Erosion Rate vs. Time for Various Nozzles

In order to relieve the compressive stress which was generated in the flange area as a result of the shrink fit provided by the adapter during assembly, this operation was discontinued. Instead, a very mild shrink fit (~ 0.005 in. in diameter) was employed and zinc chromate paste was used to accomplish sealing. The nozzle was then held in position by a retaining plate as shown in Figure 10. This arrangement was used to evaluate nozzles A-12 and C-11, neither of which failed in thermal shock.

The performance of nozzle A-2, which was pack-siliconized plasma-sprayed tungsten, was similar to that of nozzle A-8, a pack-siliconized wrought tungsten. There is some indication, however, that failure of the tungsten during burn-through may have been due to either oxidation or fracture.

Nozzle C-11, consisting of $\text{SrZrO}_3\text{-HfO}_2$, a $\text{HfO}_2\text{-W}$ grade layer, and plasma-sprayed tungsten, was fired for 51 sec before failure of the tungsten occurred. However, after 4.5 sec an abrupt sizable decrease in chamber pressure occurred indicating a marked increase in the throat area of the nozzle. Post-firing examination shows that the erosion is again localized, being confined to about 180° of the throat and convergent cone diameters. It is difficult to establish the mode of failure with accuracy; however, it appears that spalling of the inner coating or coating layers occurred after 4.5 sec of operation. Subsequent exposure resulted in rapid oxidation of the tungsten substrate and eventual burn-through. In the areas where spalling did not occur, the coating system is entirely intact, although slight microcracking of the outer oxide layer is apparent.

III. SUMMARY

A series of coated refractory metal nozzles were evaluated as radiation-cooled components in a rocket test engine. The coating systems included silicides, silver-silicide mixtures, hafnium nitride, W-Hf alloy, and graded $\text{HfO}_2\text{-SrZrO}_3\text{-W}$ composite coatings. A mixture of 50 w/o N_2H_4 and UDMH fuel was used with N_2O_4 oxidizer operating at an O/F ratio of 1.6. The nozzles were fired at sea level at a chamber pressure of 150 psia. The firing conditions were established to produce a wall temperature of approximately 4200°F at the surface of the coatings.

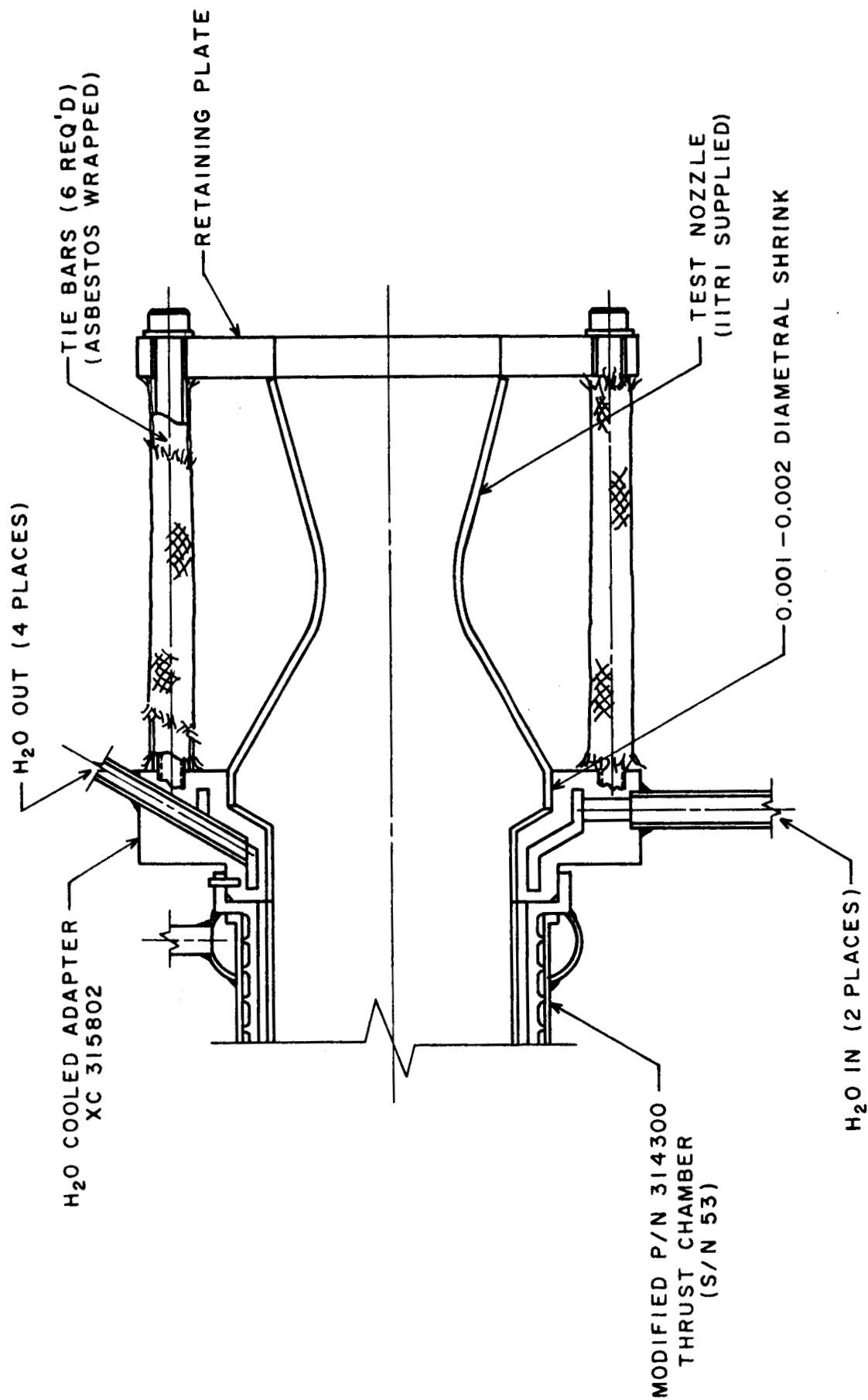
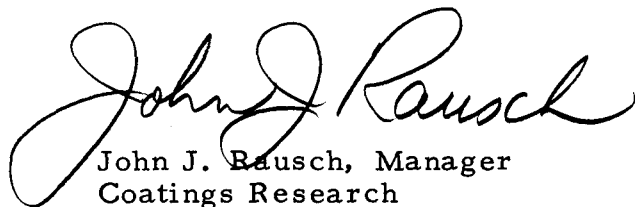


Fig. 10 - Test Installation for Refractory Metal Coated Nozzles (Showing Retaining Plate)

A pack-siliconized tungsten nozzle, which was chosen as a standard, representing the best current state-of-the-art coating system, failed after 11.8 sec under these conditions due to melting of the silicide layers. A silver-silicon diffused slurry coating showed no improvement, failing after 10 sec. An attempt to evaluate a higher melting silicide coating system, based on a modification with hafnium, was not successful due to thermal shock failure of the plasma-sprayed tungsten nozzle substrate to which this coating was applied. A coating of W_2Hf applied to a wrought tungsten substrate withstood 20.5 sec of firing prior to failure. This failure was again caused by melting.

Attempts to evaluate coating systems based on HfN and graded composite oxide coatings consisting of HfO_2 and W- HfO_2 grade layers were unsuccessful due to thermal shock failure of the nozzles. An alternate test method was devised for evaluating a composite nozzle consisting of $SrZrO_3$ - HfO_2 and W. Although gross thermal stress failure did not occur, spalling of the inner-oxide protective coating occurred after 4.5 sec of operation which eventually led to oxidation-erosion and burn-through of the underlying tungsten.

Respectfully submitted,
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